



NO. 129986 C/HO

PATENT APPLICATION
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Docket No: Q62454

Mitsuhiro Kanada, et al.

Appln. No.: 09/750,125

Group Art Unit: 1771

Confirmation No.: 6746

Examiner: Victor S. Chang

Filed: December 29, 2000

For: MICROPOROUS SOUNDPROOFING MATERIAL

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Takayuki Yamamoto, hereby declare and state:

THAT I am a citizen of JAPAN;

THAT I have graduated with a Master's degree in Engineering from Shizuoka University,
Department of Industrial Chemistry in March 1984;

THAT I have been employed by Nitto Denko Corporation since April 1984, where I hold
a position as Chief Researcher in Production Engineering Development Center of the Company;

THAT I am a co-inventor of the invention described and claimed in the above-identified
application;

THAT I am familiar with the prosecution of the above-identified application; and

THAT the experimentation set forth below was conducted by me or under my direct
supervision.

I hereby submit experimental data showing unexpectedly superior results from the claimed composite metal hydroxide. The claimed composite metal hydroxide exhibits unexpectedly superior effects as shown from the following comparative experiments which were conducted using metal hydroxides other than the claimed specific metal hydroxide.

Experiment 1 (Comparison)

50 parts by weight of polypropylene having a density of 0.9 g/cm^3 and a 230°C melt flow rate of 4, 50 parts by weight of an ethylene/propylene elastomer having a JIS-A hardness of 69, and 100 parts by weight of $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ (average particle diameter: $0.6 \text{ }\mu\text{m}$) were kneaded by means of a kneading machine equipped with roller type blades (trade name "Labo Plastomill", manufactured by Toyo Seiko Seisaku-Sho, Ltd.) at a temperature of 180°C . Subsequently, the resulting mixture was formed into a sheet having a thickness of 0.5 mm and a diameter of 80 mm with a hot platen press heated at 180°C .

This sheet was placed in a pressure vessel and held in a 150°C carbon dioxide gas atmosphere for 10 minutes at an elevated pressure of 15 MPa to thereby impregnate the sheet with carbon dioxide. After 10 minutes, the pressure was abruptly lowered to obtain an expanded material consisting of the olefin polymers. This expanded material had a degree of expansion of 1.4 times.

Experiment 2 (Comparison)

An expanded material was obtained in the same manner as in Experiment 1 above, except that $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ (average particle diameter: $8.0 \mu\text{m}$) was used in place of $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ (average particle diameter: $0.6 \mu\text{m}$).

This expanded material had a degree of expansion of 2.2 times.

Experiment 3 (Comparison)

An expanded material was obtained in the same manner as in Experiment 1 above, except that $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ (average particle diameter: $25 \mu\text{m}$) was used in place of $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ (average particle diameter: $0.6 \mu\text{m}$).

This expanded material had a degree of expansion of 6 times.

Experiment 4 (Invention)

An expanded material was obtained in the same manner as in Experiment 1 above, except that $\text{MgO} \cdot \text{NiO} \cdot \text{H}_2\text{O}$ (average particle diameter: $0.7 \mu\text{m}$) was used in place of $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ (average particle diameter: $0.6 \mu\text{m}$).

This expanded material had a degree of expansion of 33 times.

The following additional comparative experiments were conducted to demonstrate that the effect of the present invention is not obtained by the hydrated metal compounds taught in Nakae.

Experiment 5 (Comparison)

50 parts by weight of polypropylene having a density of 0.9 g/cm^3 and a 230°C melt flow rate of 4, 50 parts by weight of an ethylene/propylene elastomer having a JIS-A hardness of 69, and 100 parts by weight of hydrotalcite ($6\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$) (manufactured by Kyowa Chemical Industry Co., average particle diameter: $0.5 \mu\text{m}$) were kneaded by means of a kneading machine equipped with roller type blades (trade name "Labo Plastomill", manufactured by Toyo Seisaku-Sho, Ltd.) at a temperature of 180°C . Subsequently, the resulting mixture was formed into a sheet having a thickness of 0.5 mm and a diameter of 80 mm with a hot platen press heated at 180°C .

This sheet was placed in a pressure vessel and held in a 150°C carbon dioxide gas atmosphere for 10 minutes at an elevated pressure of 15 MPa to thereby impregnate the sheet with carbon dioxide. After 10 minutes, the pressure was abruptly lowered to obtain an expanded material consisting of the olefin polymers. This expanded material had a degree of expansion of 5.6 times.

Experiment 6 (Comparison)

An expanded material was obtained in the same manner as in Experiment 5 above, except that basic magnesium carbonate ($3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$) (average particle diameter: $6\text{ }\mu\text{m}$) was used in place of hydrotalcite ($6\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$).

This expanded material had a degree of expansion of 1.2 times.

EVALUATION

Flame retardancy of the expanded materials obtained in Experiments 1 through 6 above was evaluated in the same method as described on page 32 of the specification of the present invention.

The degree of expansion was determined by the following equation.

$$\text{Degree of expansion} = (\text{density of sheet before expansion}) / (\text{density of expanded material}) \\ = 1 / (\text{relative density}).$$

The results obtained in Experiments 1 through 6 above are shown in the Table below together with the results obtained in Examples 7, 8 and 9 of the present application.

| | Metal hydroxide (Average particle diameter) | Flame retardancy | Degree of expansion (Times) |
|--------------|---|------------------|-----------------------------------|
| Experiment 1 | Al_2O_3 (0.6 μm) | Unacceptable | 1.4 |
| Experiment 2 | Al_2O_3 (8.0 μm) | Unacceptable | 2.2 |
| Experiment 3 | Al_2O_3 (25.0 μm) | Unacceptable | 6.0 |
| Experiment 4 | $\text{MgO}\cdot\text{NiO}\cdot\text{H}_2\text{O}$ (0.7 μm) | Acceptable | 33 |
| Experiment 5 | $6\text{MgO}\cdot\text{Al}_2\text{O}_3\cdot\text{H}_2\text{O}$ (0.5 μm) | Unacceptable | 5.6 |
| Experiment 6 | $3\text{MgCO}_3\cdot\text{Mg}(\text{OH})_2\cdot 3\text{H}_2\text{O}$ (6 μm) | Unacceptable | 1.2 |
| Example 7 | $\text{MgO}\cdot\text{ZnO}\cdot\text{H}_2\text{O}$ (1.0 μm) | Acceptable | 25 |
| Example 8 | $\text{MgO}\cdot\text{ZnO}\cdot\text{H}_2\text{O}$ (1.0 μm) + α | Acceptable | 13 |
| Example 9 | $\text{MgO}\cdot\text{ZnO}\cdot\text{H}_2\text{O}$ (0.5 μm) | Acceptable | 19 |

α : Ethylenebispentabromodiphenyl

Experiments 1-3 and 5-6: Comparison

Experiment 4 : Invention

As is apparent from the results shown in the above Table, use of the specific metal hydroxide in a microporous soundproofing material can satisfy flame retardancy of the material and can also remarkably increase a degree of expansion of an expanded material.

The unexpectedly superior results are attributed to the following. In a production method of an expanded material using an insert gas as an expanding agent as in the present invention, the insert gas vaporizes together with rapid pressure drop, resulting in growth of pores. In such a

case, if affinity between a resin and a flame retardant is poor, the resin peels at the boundary, resulting in formation of vent holes of the inert gas. As a result pores do not grow.

Contrary to this, the metal hydroxide has good affinity with a resin. Therefore, a resin fluidizes well, and pores grow. As a result, it is considered that a high degree of expansion is obtained.

Expanded materials having a very high degree of expansion have small compression stress and conform well to a rough surface, so that high soundproofing property is achieved, as described in the present specification. Therefore, such materials are suitable as a soundproofing material.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: Aug. 122/2005


Takayuki Yamamoto (co-inventor)